THIRD QUARTERLY PROGRESS REPORT

RESEARCH and DEVELOPMENT in Cds PHOTOVOLTAIC FILM CELLS

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FORWARD

The work of this project has been carried out at the Crystal-Solid State Research Laboratory of the Harshaw Chemical Company at 2240 Prospect Avenue, Cleveland, Ohio. Project direction has been provided by Mr. F. A. Shirland. Mr. T. A. Griffin has acted as the Project Supervising Engineer and Principal Investigator.

The following personnel have contributed to the work of this Contract and charged time approximately as indicated during the period December 1962 through February 1963.

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Miscellaneous Analy	tical, testing and shop labor		154		
		Total	2046 1		

It is estimated that the work of the first nine month period of this project is 100% complete.

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INTRODUCTION AND SUMMARY

This report describes the work performed during the third quarter period of a one year research and development contract on CdS thin film photovoltaic cells sponsored by the Lewis Research Laboratory of NASA with Dr. Andrew E. Potter, acting as Contract Monitor.

During this period, an evaporator was tooled for the purpose of evaporating uniform CdS films on 6" x 6" substrates. Several large area films were made, some of which were completed as front wall photovoltaic cells. Two of these large area cells were about 2.0% in efficiency. These 36 in² cells are believed to be the largest single photovoltaic cells ever made. The films that these cells were fabricated from were originally intended only for physical measurements e.g. uniformity of thickness, adherence, crystallite size, etc.

The average efficiency of the standard pilot line dropped to 2.06% with a corresponding increase in the scrap rate. This drop in efficiency was attributed to several problems that have subsequently been corrected. The maximum efficiency during this period was 3.5%.

More experiments on the effect of substrate temperature were run with a tighter control of film thickness. These experiments confirm the previous data concerning the major changes in film resistivity. An attempt will be made to further relate the bulk resistivity of the evaporated films to cell operation.

Measurements were initiated to determine the mechanism of the apparent sheet resistance increase caused during lamination.

Tungsten was used successfully as a substrate material.

PILOT LINE

During this three month period, two hundred fifty-eight cells were produced on the pilot line with an average efficiency of 2.06% and a maximum efficiency of 3.5%. Sixty-seven cells were adjudged as scrap.

One of the causes for the lower average efficiency was the production of less uniform films. This lack of uniformity of the films was found to be caused by a general build-up of evaporant on all the surfaces within the vacuum bell jars. Immediately after the systems were completely cleaned, the conversion efficiencies of completed cells rose sharply.

Another problem was traceable to an incorrectly reading thermocouple. It appears that the vapors in the vacuum system gradually attack the thermocouples, and unless they are changed regularly they tend to give faulty readings. This caused erroneous substrate temperature readings resulting in many too-thin films. These thin films tend to develop shorts during barrier plating.

The third problem encountered was a material problem. Some of the poor cells can be traced to films made from certain sinters of CdS. One lot was not completely sintered because of a power failure during the run. Other sinters display high

oxygen content. More rigid control of the material used for evaporation, has been established.

The pilot line has continued to serve as a base line of operation and as a source of cells for experiments.

EVAPORATION EQUIPMENT

1. Substrate Heater

The main effort in the second six month period was to retool the evaporator to enable the handling of larger substrates, toward the goal of producing 6" x 6" cells.

The first retooling efforts were directed toward the substrate heater. Formerly, radiation type molybdenum heaters were placed about one inch above the substrate, but because of inefficient heat transfer it was decided to change to a conduction type heater. The new substrate heater chosen was an immersion heater enclosed in an incoloy sheath. Figure 1 shows the substrate heater and holder. It has an 11" diameter circle parallel to the base plate. The major length $(27\frac{1}{2})$ " of the 33" circumference) of this circle contains a 1.5 k.w. heater. The cold ends of the heater are $28\frac{1}{4}$ % long, act as the vertical supports, and as feed-throughs on the base plate. The bulkhead seals are made with flexible conduit seals as can be seen in Figure 2. As a result of this construction, the substrate heater is both self-supporting and adjustable.

Figure 1 clearly shows that the substrate heater is clamped tightly to the substrate holder. The original design of the holder consisted of a 1/8" aluminum circular plate about 12" in diameter, but because of excessible warping, it was changed to a $\frac{1}{4}$ " aluminum plate. The substrates, which are slightly larger than the 6" x 6" square hole in the center of the substrate holder, cover the opening and are backed up by another aluminum plate. This back-up plate is also bolted to the substrate holder. In order to improve the heat transfer, the back of the substrate and the front of the back-up plate are covered with a black coating.

Originally, four bolts were used to secure the back-up plate and the substrate. Now, however, eight are used to minimize the effects of warping of the whole substrate system due to the high temperature.

The ease with which the substrate to filament distance is adjusted makes this heater very versatile. Initially because of the difference in the thermal expansion of the molybdenum substrate and the aluminum holder some buckling was produced in the substrate during evaporation. This suggested that the aluminum be replaced by another metal with an expansion coefficient closer to molybdenum, until it was discovered that the buckling was almost completely overcome by using a graphite coating between the substrate and the substrate holder. This allows the two metals to slide against each other.

This substrate heater is much more efficient than the radiation type since it requires about one fourth the amount of power to heat the same area. During the on-cycle, about 2 k.w. is used to heat the 6" x 6" area, whereas 1 k.w. is also required to heat the 3" x 3" areas employing a radiation heater. In general, it can be said that this heater has proven to be very satisfactory for heating the

large area substrates.

Filaments

The filaments used for the evaporation of the CdS on 6" x 6" substrates is the four boat configuration used on the standard pilot line(1). The boat size had to be increased to handle four times the volume of charge since the film area has been increased that amount. The position of the boats was adjusted to obtain uniformity of deposit over the 6" x 6" substrate area. The tantalum filaments used at present are $\frac{1}{2}$ " in diameter and $3\frac{1}{4}$ " long. The four filaments are on 4" centers, and the substrate to filament distance is 9.2". The basic procedure used for achieving uniformity is to place the filaments on centers equal to the substrate length, in this case, on 6" centers. Then the substrate to filament distance is adjusted to 75% of the six inches. (2) This basic method does not hold completely true here because the filaments are somewhat collimated sources. The final distances arrived at are adjustments made by trial and error.

The filaments are wired in series because this arrangement has advantages over the former parallel, or series parallel installations. These advantages are, firstly, individual filaments can be changed without disturbing the other three. Secondly, and most important, the line drop is minimized permitting the power to be more efficiently coupled to the filaments. It has been found that only about one half of the originally estimated power, or a little more than 2 k.w. is required at present. Figure 3 shows the filament arrangement. The charge used in the filaments is 22 grams per filament which is about four times the amount used for evaporation on 3" x 3" areas. The uniformity of the films produced will be discussed below.

In order to conserve power and also reduce the heat radiated to the bell jar, tantalum reflectors are used on the outside of the filaments. These shields are incorporated as part of the tantalum caps that serve to keep the charge from flying out of the boats during evaporation.

In an attempt to eliminate all possibilities of spattering, quartz plugs were placed in the open end of the filaments. These plugs greatly reduced the spattering, the evaporation rate, and hence the film thickness. Additional experiments to determine the feasibility of this approach must be deferred until the larger power supply arrives, since the present supply was operating at 95% to 100% of peak power.

Considerable heating of the bell jar, the base plate, and feed throughs has been noted, but no ill effects, such as damaged seals, have resulted. As yet it has not been deemed necessary to incorporate a cooling system within the bell jar. One immediate advantage a cooling system would offer is a shorter cooling time before the system could be opened to air.

EVAPORATED 6" x 6" FILMS

During this period, eleven large area films were evaporated after the present filament spacing and distance was established. As a test of uniformity two films had detailed thickness measurements taken. The first film had the CdS removed from the substrate in nine positions and the thickness measured. The CdS thickness was found to vary from 1.5 to 2.1 mil. Then all the CdS film was stripped off, collected

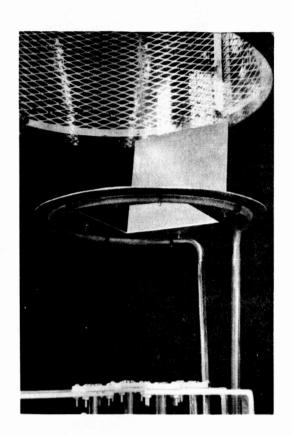


Figure 1
SUBSTRATE HEATER AND HOLDER.

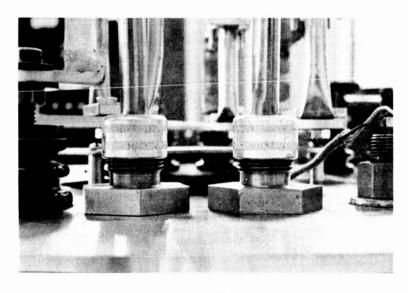


Figure 2

BULKHEAD FEEDTHROUGHS
FOR SUBSTRATE HEATER.

and weighed. On the basis of total weight and the area covered, the thickness was found to be 1.7 mil. A second film had its deposit measured by using a microscope to measure the thickness of the CdS at the edges of several holes placed in the film. The thickness was found to vary from 1.7 to 2.1 mils by this method. Since these types of measurements are destructive, subsequent films were only checked with a surface gauge. The uniformity seemed reasonable and sufficient for the production of good films. About 8% of the total filament charge was collected on the substrate. If it is desired to collect more of the charge, the rate of evaporation must be increased. The explanation for this is described elsewhere. (3)

FABRICATED 6" x 6" CELLS

Of the eleven films evaporated under the conditions described above, two were destroyed in the measurements of deposit thicknesses, three were too thin, two suffered from cracking or poor adherence, and four were made into cells. One of the latter four cells was shorted. The other three had efficiencies of about 2.0% and their properties are described in Table 1. These results are very encouraging for so few attempts, particularly since the efficiencies are almost as high as the pilot line average results. This is especially heartening since the CdS material used was considered poor material since it seemed to produce low efficiency cells when used on the pilot line. It was used here since the physical properties such as thickness, crystallite size, etc; were the properties that were of first interest, and high cell conversion efficiencies were only of secondary interest. Better material will now be used in future efforts. Figure 4 shows one of the completed 6% x 6% cells.

During the next quarter the properties of the 6" x 6" cells will continue to be studied in an effort to see what improvements can be made. The effect of some properties e.g. series and sheet resistance, will probably be exaggerated because of the larger area which, in turn, should facilitate an investigation of the causes and importances of these, and possibly other properties.

One fact that is immediately apparent when comparing the 6" x 6" cell with a 6" x 6" array made of four 3" x 3" cells is the active area gained. It amounts to more than 10%.

A larger pressure test unit is being constructed similar to the one reported in the last quarterly report. (4) This obviates the need for laminating each cell, and yields performance data while reducing time and cost.

EVAPORATION PARAMETERS

In the last quarterly report data was supplied on some work done to evaluate the parameters of CdS evaporated films. (4) A difference was found in the electrical properties of cells evaporated at different substrate temperatures. The fact that the thickness of the films used in the study varied greatly (from 2.5 to 0.6 mils.) cast some doubt on the results. This series was repeated and an effort was made to control the thickness of the films. As seen in Table 2, the thicknesses were held within the range of 0.8 to 1.0 mil. These are on 1" x 3" cells.

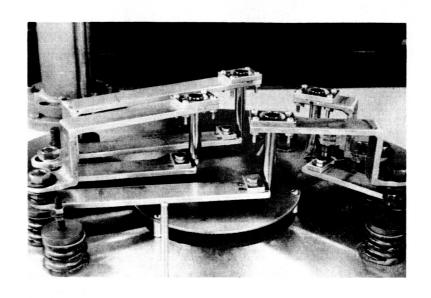


Figure 3

FILAMENT ARRANGEMENT FOR 6" x 6" FILM EVAPORATION.

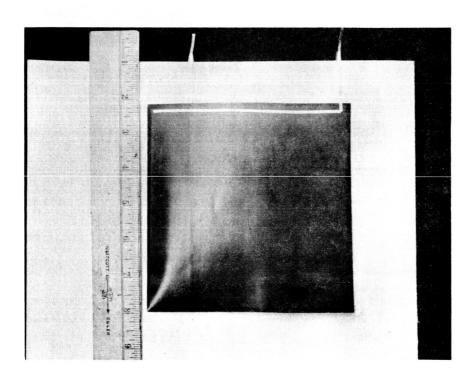


Figure 4
6" x 6" CELL

TABLE I

PERFORMANCE OF 6"x6" FRONT WALL CdS FILM CELLS

ARRAY NO.	CELL NO.	PLASTIC	OCV (volts)	SCI (Amps)	AREA (cm ²)	PWR. (m w)	EFF. (%) (_£	WT. grams)	W/1b. @ 140 mw/cm ²	SUBSTRATE COLLECTOR
371 MN	5	Mylar Nylon			·SHORTED-					0.002" Mo. 70 l/in Au
384 MN	7	Mylar Nylon	0.47	> 2.0*	203	410	2.02	18	14.5	0.002" Mo. 70 l/in Au
385 MN	8	Mylar Nylon	0.45	1.96	200	3 60	1.8	20	11.5	0.002" Mo. 70 l/in Au
406 MN	11	Mylar Nylon	0.46	> 2.0	204.3	418	2.04	16.6	16	0.002" Mo 70 l/in Au

^{*} The short circuit current could not be measured with the present test equipment. A transformer with a higher current carrying capacity is required.

The same CdS material was used throughout, and the only parameter that was varied was the substrate temperature. Again in this series of tests, as in the last series, the films evaporated at the standard substrate temperature display the highest resistivity. Again the highest open circuit voltage for the processed cells was found in cells made from films evaporated at substrate temperatures higher than the standard setting of 220°C.

In this series of tests, the temperature reached was not as high as in the previous series due to the limiting factor of substrate heater power. Yet, all the films evaporated at temperatures above the standard operating temperature in both series show a decrease in resistivity. The cells made from these films also exhibit a decrease in current density.

This whole question of bulk resistivity and how it effects the final cell output is worth examining. First of all, it should be pointed out that the material evaporated is undoped sintered CdS. Therefore, any variations in resistivity probably comes from non-stoichiometry. This is evident in the low resistivity films achieved by evaporation at low substrate temperatures. These films are very dark, which indicates a large cadmium excess as the cause of the high conductivity.

Pertinent to this question are some series resistance calculations made on CdS film cells. The I-V curves of the 6" x 6" cells show a series resistance of about 0.1 ohm while the 1" x 3" cells show about 1 ohm. Comparing the two sizes on a basis of area and thickness, the bulk resistivity of the CdS film could be the cause of this series resistance. Calculations for the one ohm series resistance of one particular 1" x 3" cell resulted in a bulk resistivity of 3 x 10³ ohm-cm; while the one-tenth ohm series resistance on a 6" x 6" cell yielded a bulk resistivity of 5×10^3 ohm-cm. These films were evaporated at a substrate temperature of 220°C. As seen from Table 2, bulk resistivities in the 10³ ohm-cm range would be expected for the substrate temperature at which these films were evaporated. Whether this cell series resistance can be related to the bulk resistivity, or not, is to be determined. However, the data does seem to fit, and if this is true, then this may well be an area for great efficiency improvement. The single crystal cells that reached a reported 6 and 7 per cent efficiency were made from one ohm centemeter indium-doped material which seems to support this idea. Some effort will be made to check on this possibility. It shall be interesting to learn the variations in cell operation between cells made from films with different bulk resistivities caused by doping, and cells made from films with different bulk resistivities caused by non-stoichiometry.

This work shall be initiated using some doped single crystal material as the evaporant.

SHEET RESISTANCE

A study of the sheet resistance of the "barrier" during this quarter was necessitated by the results of experiments in the first two quarters. During the first quarter, (5) the sheet resistance was measured experimentally, to be about 10 ohms per square. An increase in sheet resistance has been noted after lamination. Calculations indicate that a collector mesh of 20 lines per inch would be adequate assuming a barrier sheet resistance of 10 ohms per square. During the second

TABLE II

PROPERTIES OF FILMS EVAPORATED AT VARIOUS SUBSTRATE TEMPERATURES

UBSTRATE EMPERATURE (°C.)	RESISTIVITY (in dark 1 hour) (ohm-cm)	RESISTIVITY (in light 150' candlæ)(ohm-cm)	CELL NO.	THICKNESS (MILS.)	OCV (Volts)	SCI (ma.)	EFF. (%)
140	375	200	х479 х480	1.0 1.0	0.42 0.5	12 58	N.C.* 1.3
180	65 0	340	х476 х477	0.8 0.9	0.48 0.51	36 65	0.8 1.1
220	5200	1300	X48 3 X484	1.0 1.0	0.49 0.5	124 116	2.2 2.4
250	1300	330	X471 X472	1.0 1.0	0.52 0.52	124 120	2.8 2.6
260	850	230	x468 x469	0.9	0.5 0.5	104 88	2.0 1.8

Material (CdS) was from same sinter. (0-122)

Resistivity measurements made on pyrex slide that was evaporated at same time as films on molybdenum.

*N.C. = Not calculated - very low.

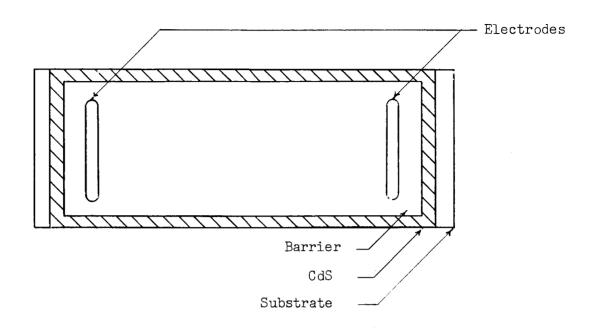


FIGURE 5

TEST CELL ELECTRODED FOR SHEET RESISTANCE MEASUREMENTS

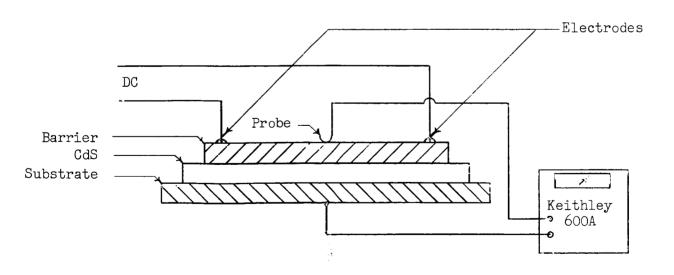


FIGURE 6

CIRCUIT USED TO SHOW HOW CURRENT LEAKS INTO THE SUBSTRATE

quarter, experiments with the pressure test unit gave results which were consistent, within experimental deviation, with the calculations. At the present, the increase of sheet resistance with lamination remains a possible limiting factor on the grid spacing in completed cells.

Before a number of tests were run to pinpoint this apparent sheet resistance increase, certain criteria were used in the selection of the sample cells. They all were chosen from cells evaporated on 1" x 3" x 0.002" molybdenum substrates from the same batch (0-93) of CdS material. They were all evaporated in the same evaporator with a substrate temperature of 220° C, a source temperature of 1060° C. The duration of evaporation was 20 minutes. The film thicknesses were chosen as 1.27 mils. or thicker. Cell efficiencies were 1.60% or higher. The cells finally selected were the unlaminated cells:

S-278	W-408
S-289	W-417
W-406	W-418
W-407	W-420

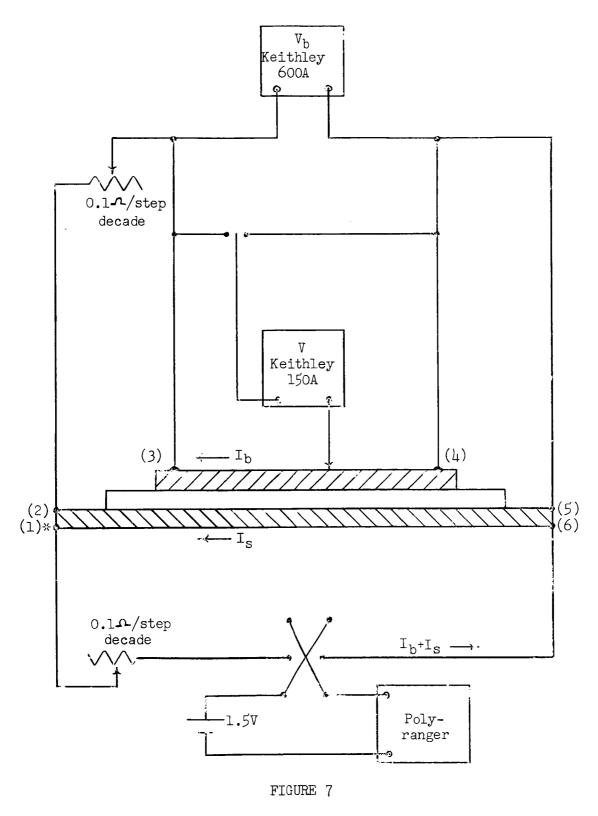
Laminated cell #668 was used in setting up the sheet resistance measuring equipment.

The circuit used to measure the sheet resistance of the barrier was shown in the first quarterly report. (5) An important feature of the test method is an attempt to match the potential of each point on the "barrier" with the potential of the point on the substrate directly beneath it. Because of the resistance of the n-type cadmium sulphide layer between the "barrier" and the substrate, small differences in the potentials should not introduce appreciable errors in the sheet resistance measurements. It was found that reversing the power source gave approximately the same value for sheet resistance. Reversing the cell itself, in certain cases, gave a different value for sheet resistance. In some cases, the difference was as great as a factor of two. The assymetry of the measurements for these cells probably results from large mismatches in the potential distributions of the "barrier" and the substrate. Figure 5 represents a typical test cell, electroded for sheet resistance measurements.

To substantiate the method of measuring the sheet resistance, the surfaces of some of the cells were probed in order to get a picture of the potential distribution. Figure 6 is a schematic diagram of a circuit used to demonstrate how current leaks through the cadmium sulphide to the substrate when current is fed only into the electrode stripes on the "barrier". A loop of fine wire was used as a probe in these measurements. As expected, the equipotential lines were crowded together near the stripes, indicating conduction to the substrate.

Figure 7 represents a circuit used to explore the potential distribution of cells under the conditions of sheet resistance measurement. It is similar to the one shown in the first quarterly report (5) except that a microvoltmeter is used with the surface probe.

To facilitate equipotential tracing, the cell should be relatively immobilized. Several attempts to accomplish this by holding the cell against an evacuated plate were unsuccessful.



CIRCUIT USED TO EXPLORE THE POTENTIAL DISTRIBUTION OF CELLS

* Numbers in parentheses refer to electrical contact positions (See Figures 8 & 9, Page 14).

A plastic "splint" was used to help immobilize the cells during probing. The cells and the splint were held by rubber bands to a small metal plate attached to a ringstand. The splint was specially notched to permit access for attaching clips to the substrate.

When a cell, which gave nearly symmetrical resistance tests results, was probed in the usual test circuit the mid-line potential distribution was essentially linear. A rough check of a few equipotentials gave approximately the expected results.

Contact to the substrates of unlaminated cells measured during the first quarter was made with "microgator" clips along one edge of the substrate. The potential distribution on a substrate under such conditions was determined by probing, and is shown in Figure 8.

Many adjustments and measurements were required during electrical potential probing. Almost every adjustment and measurement required undraping and redraping of the sample and probe with black cloths. Since the cloths were not completely opaque and the set up was irregularly shaped, it was not certain whether there was any detrimental light leakage. For this reason, the sample holding apparatus, the probe, and its x,y,z drive were reinstalled in a dark box.

An unlaminated cell with the usual microgator clip arrangement was probed using the dark box arrangement to show "contact resistance" between the clips and various parts of the cell. The contact resistance was noticeable but not large enough to contribute much error to the measurements.

The wire loop probe was replaced by a "snail" (6), a small cylinder of conductive elastomer, approximately 0.1 cm in diameter. This greatly reduced fluctuations in potential readings.

Figure 9 illustrates a potential distribution of the face of a cell including the edge of the substrate. Such a cell has approximately the same sheet resistance when it is reversed as when first tested. Other cells had distorted potential distributions and gave a different value for sheet resistance depending how they were placed in the circuit. That was probably due to localized leakage in the barrier. These cells will be eliminated from the series of tests on sheet resistance. The above results indicate that the amount of mismatch in the potential distributions of the barrier and substrate in symmetrical cells is not enough to introduce appreciable error.

Plans for the future include selecting more cells by criteria similar to those mentioned above and then eliminating those which show a different sheet resistance when reversed in the circuit. This should cull out those with leakage paths between the barrier and substrate.

Several areas will be investigated in an effort to locate the cause of the apparent sheet resistance increase under laminating conditions. The possibility that the laminating procedure effects the ohmicity of the metal contact to the barrier and consequently the sheet resistance will be explored. It is planned to make a determination of the change of sheet resistance with lamination. Study of the possible effects of different plastics will also be made.

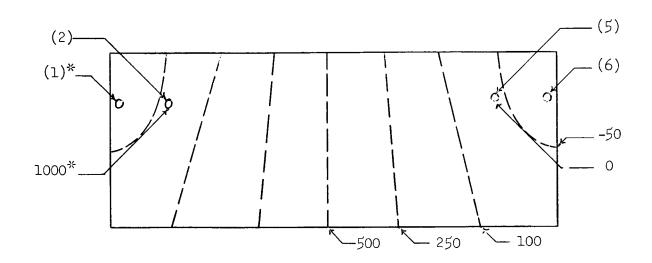
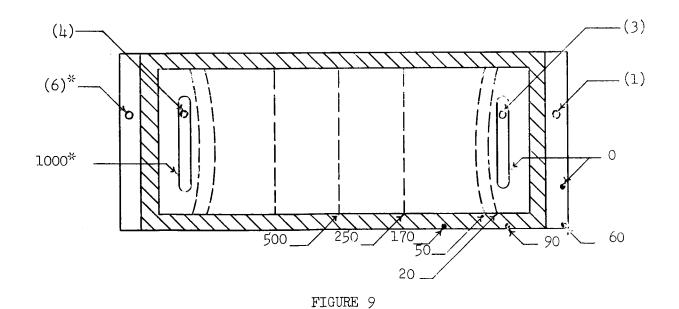


FIGURE 8
POTENTIAL DISTRIBUTION ON MOLYBDENUM SUBSTRATE



POTENTIAL DISTRIBUTION ON FRONT SURFACE OF CELL

* Numbers in parentheses refer to electrical contact positions (See Figure 7, Page 12). Plain numbers indicate electrical potential in micro-volts.

SUBSTRATES

Presently the work with the 6^{tt} x 6^{tt} films will be done on the standard two mil molybdenum since this is the substrate of greatest reliability. However, efforts are continuing to find other materials that may prove to be of equal, or greater merit. Some of the experiments conducted in this area shall be mentioned briefly.

During this period CdS was evaporated onto one mil tungsten and it was immediately apparent that adherence was a problem. However, when the tungsten was etched prior to evaporation, a cell of slightly over two per cent was produced. Although tungsten is very dense 19.3 g/cm 2 , (almost twice as dense as molybdenum) the thermal match between CdS and the tungsten apparently was better than between CdS and molybdenum since the cell remained flat and did not curl. More work is planned using this substrate.

Some two mil niobium sheet that was produced by ordinary rolling and by Sendzimir processing was obtained and used as substrate material. No cleaning, etching, or surface conditioning used as yet has promoted any adherence of the CdS film to the niobium.

Since invar "36" had given some very good cells initially, additional attempts to repeat these results were made, but have proven rather disappointing. A high series resistance appeared in the cells. The cause of which has not been determined as yet. It appears that a film is formed on the invar during the etching procedure which has a great effect on the final results. This work is continuing.

Some "filohm" was also obtained as a possible substrate material. It consists of one mil mylar with a transparent gold layer on the surface with a resistance of about 25 ohm/sq. This material is not suitable as a substrate since it can not withstand the evaporation conditions. It was used, however, on the pressure test unit to replace the metal mesh, but the results were negative. The cells tested with it showed very low current and high series resistance. There is a possibility that it still might be used in combination with the mesh electrode.

WORK PLANNED FOR NEXT PERIOD

During the next quarter the tooling used to produce $6" \times 6"$ area films will be improved where necessary. The properties of these $6" \times 6"$ films will continue to be studied, as well as the properties of the cells made using these large area films. An attempt will be made to correlate the bulk resistivity of the films to cell operation.

The search for new and better substrates will continue.

The collector contact will continue to be studied. The work to determine the mechanism of apparent sheet resistance increase under laminating conditions will continue.

The standard process pilot line will continue to be operated to provide a base line and furnish cells for other experiments.

Near the end of this period, CdS front wall solar cells demonstrating improvements in cell size, specific power-to-weight ratio, and cell efficiency will be fabricated and delivered to the contract monitor.

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- 6. Pylon Company, Inc. Attleboro, Massachusetts.